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INTERDISCIPLINARY RESEARCH ON THE  
NATURE AND PROPERTIES OF CERAMIC MATERIALS

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### SUMMARY

This document is a report covering interdisciplinary research at the University of Washington on the nature and properties of ceramic materials for the period June 16 through December 15, 1979. The program is primarily supported by the National Aeronautics and Space Administration through Grant Number NGL 48-004-002.

The principal direction of the research is towards improvement in design with brittle (ceramic) materials through studies in design methodology and associated supportive research. The truly interdisciplinary nature of the program is evidenced by the participation during the six-month report period of 17 faculty, 14 graduate and 2 undergraduate students representing seven disciplines within the University.

N81-15105

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## INTRODUCTION

In 1963, the University of Washington inaugurated an interdisciplinary program on the nature and properties of ceramic materials supported by Grant Number NGL 48-004-002 from the National Aeronautics and Space Administration. The original proposal suggested that the research study the effects of various environments upon these classes of materials. Since its inception, the major thrusts have varied to include research on reactive metal-oxygen-carbon system, ion conduction ceramics, processing and the effects of mechanical stress. These modifications have been the result of changes in faculty interests and rapid technological advances. In early 1976, participating faculty agreed upon a redirection of the program toward the understanding of and improvement in the utilization of ceramic materials for structural applications in severe environments. This was in keeping with our original proposal, dated March 14, 1963, which stated that "materials development for energy conversion systems...will be studied...for potential high temperature use...and that structural design investigations will be undertaken guided toward the utilization of...ceramics (since) no known attempts have been made to build any thermal energy conversion device entirely, or to the greatest possible extent, from brittle-failure type materials".

The previous development of interdisciplinary communications and the interest and dedication of faculty members with the necessary expertise have resulted in satisfactory progress toward our stated goals, which are:

1. Advancement of material performance and design methodology as related to brittle materials.
2. Understanding of processing and properties of ceramic materials as related to design requirements.
3. Improvement of communication and understanding both within the University and between academia, government, and industry.

The program supported by this grant is divided into two major components--studies of design methodology and structural ceramics research. The purpose of the former is not only the study, improvement and utilization of probabilistic design but also the development of means for establishing the necessary communication and mutual understanding between the designer and the materials specialist. The research effort consists of interrelated studies of processing, characterization and mechanics, including testing and evaluation. Each of the two major areas are coordinated by a committee composed of faculty members active in that area. Faculty, staff and students participating in this program are listed in Appendix I. The administrative responsibilities of this program have been assigned by the University to the Dean of the College of Engineering, who appoints a board of representative faculty members to establish policy and approve the general operation of the program. The composition of the Ceramic Materials Research Committee is shown in Appendix II.

## OVERVIEW

Progress toward the first two goals will be discussed in the following sections of this report dealing with design methodology studies and structural ceramics research. Achievements toward the third goal, during this report period, included the annual program review; the presentation of mini-reviews of the program for personnel from government, industry and universities; visits to installations involved in development of high performance ceramics for structural applications and attendance at professional meetings. The mini-reviews were given by participating faculty members in total to about 55 persons at the Boeing Company and Tektronix, Inc. In addition, a similar presentation was made to the College of Engineering Visiting Committee at its December meeting.

Installations visited included Ames Research Center, Kennedy Space Center, Naval Research Laboratory, Army Materials and Mechanics Research Center, Eaton Corporation, Ford Motor Company, General Motors Technical Center, Kyocera International, Inc., Lockheed Missile and Space Corporation, Norton Company, Rockwell International, Inc., and Tektronix, Inc. Foreign organizations visited included Daimler-Benz, MTU, Annawerk and Rosenthal in Germany; United Turbine in Sweden and Ishikawajima-Harima Industry, Tokyo Shibaura Electric Company Research Laboratory, and Japan Steel Company in Japan. Technical meetings attended were the Third Chemical Conference, Vancouver, British Columbia; the American Vacuum Society, New York City and the Third International Conference on Mechanical Behavior of Materials, University of Cambridge, U. K. The participants in this program were instrumental in the arrangements for and paper presentations in a two and one half day session entitled "Design with Brittle Materials" during the Pacific Coast Regional Meeting of the American Ceramic Society held in Seattle. The program for this session is given in Appendix III.

The annual review of this program was held on campus July 9. In addition to the NASA technical monitor and his committee, a total of 40 invitees representing industrial and government organizations attended. This substantial number was attributed to scheduling the review on the day prior to the AMMRC meeting at East Sound, Washington. A list of those present at the program review will be found in Appendix IV.

Dr. Alvin E. Gorum, Research Professor of Ceramic Engineering and coordinator of the supportive research portion of this program, resigned from the faculty in September to accept a position in industry. He will continue to hold an appointment as an Affiliate Professor. His departure will be keenly felt by faculty and students alike as his knowledge, advice and assistance played a significant role in the total program.

An opportunity for industrial affiliation in this program was announced last spring and we were pleased to welcome the Eaton Corporation as the initial member.

In striving to develop a center of excellence in design with brittle materials with emphasis on high performance ceramics, we have sought additional funding to supplement the NASA grant. In addition to the previous contracts from the Fossil Fuel Division of the Department of Energy, contractual support was received from the Department of Army and Compustion Research & Technology, Inc. Successes achieved in the overall program have been due to the interest and cooperation of a very dedicated group of faculty members, and to the encouragement offered by our colleagues in government and industry.

## DESIGN METHODOLOGY STUDIES

### Coordinating Committee

R. J. H. Bollard, Aeronautics and Astronautics, Chairman  
B. J. Hartz, Civil Engineering  
A. S. Kobayashi, Mechanical Engineering  
J. I. Mueller, Ceramic Engineering  
W. D. Scott, Ceramic Engineering  
R. Taggart, Mechanical Engineering

### Purpose

It is the intent of this portion of the program to develop institutional and individual expertise in the design of structural components utilizing ceramic materials, especially in those applications where the brittle material is exposed to hostile environments.

### Objectives

1. Continue the offering of the instructional sequence of 476-496 and 536 to qualified undergraduate and graduate students.
2. Develop recommended changes and additions to the first edition of the workbook "Design with Brittle Materials."
3. Develop supportive illustrative materials for both the workbook and the monograph prepared for use in the sophomore and junior level mechanics of materials courses.
4. Develop a compilation of design projects suited to the courses 496 and 536 through discussions with industrial and governmental laboratories.
5. Develop a compilation of design study suggestions for unconstrained individual or small group projects in 496 and for graduate program focus in 536.
6. Expand the interface between the research and instructional development programs.
7. Identify problem areas which, though beyond the constraints of the class, might be developed in a manner such that the students could play a supportive and learning role.
8. Maintain the external information presentations and visits to allow more potentially interested organizations and prospective students to take advantage of the program.
9. Initiate the development of suitable instructional materials for workshops, short courses and extramural instruction.

### Summary of Current Status

Established components of the program are:

- (a) The offering of courses 476, 496 and 536;
- (b) The introduction of lower division course material in brittle materials;
- (c) Informational seminars to industry and government on the content and results of the program;
- (d) Faculty and graduate studies of meaningful real-life problems in designing with brittle materials;
- (e) The continued interaction between the research and the design methodology areas of the total program.

### Educational Development

The course sequence continues to exhibit growth and is now sufficiently popular to require limitation on enrollments in the courses 476 and 496. While the population is primarily undergraduate, the courses are open to graduate as well since these courses have proven to be very beneficial to the graduate students in the research program as they develop a broader perspective of the potential use of and the needs in research for ceramic materials. The mix of students from the participating departments remain reasonable and this is due, in no small measure, to the publicity in the lower division classes through program description material and the course work material offered to the instructors in courses dealing with materials and design.

The student workbook, titled "Design with Brittle Materials", was completed and published in July and is now in use in the current offerings of 476 and 496. The lecture notes, from which this publication version was compiled have been evolving over the prior two years and pre-publication copies were circulated to industry and government for critiquing and input. The table of contents is given in Appendix VI. The workbook is in paper-back with plastic comb binding and is available at the publication cost of \$60 per copy. A request for purchase is included as the last page of this report.

#### Course 536 - R. Taggart, Mechanical Engineering, Coordinator

The only significant changes in the course material during this report period have taken place in the graduate level project courses 536.

Summer: Probabilistic design methodology was utilized to evaluate a ceramic oxygen sensor component in the fuel injection system of 1981 series automobiles where stoichiometric combustion is an objective for increasing combustion efficiency. This study was made possible through the cooperation of the A. C. Sparkplug Division of the General Motors Corporation. The graduate students participating prepared a report which was presented before the faculty and representatives from A. C. Sparkplug and Detroit Diesel Allison. The report was subsequently presented at the "4th Annual Conference on Composites and Advanced Materials", January 20-24, 1980, Cocoa Beach, Florida, and is to be published as a portion of the conference proceedings.



Autumn: A study of design modifications in glass containers was undertaken to evaluate probability of failure methodology. This project is enhanced by the opportunity to observe the type, size and location of various flaws in the glass and to determine their effects upon failure of brittle materials. It is hoped that this study will result in an improved insight into the effect of flaws in opaque brittle materials. This project is continuing.

### Industrial Seminars and Workshops

Two industrial seminars were given during the report period and were followed by discussions and facilities tours by the faculty group. The seminars themselves have been well received and the post-seminar tours have been most helpful in identifying design study ideas for the 496 and 536 design courses. The backlog of graduate-level study projects has been developed primarily from this source and now the identification of suitable projects is, for all practical purposes, self-sustaining by inputs from these initial contacts and formal affiliations as well as from the independent studies being carried out in the program.

Three short courses and workshops in the Methodology of Design with Brittle Materials are tentatively scheduled during the next 12 months. The short course will be a 4-day coverage of current and potential applications of various brittle materials, the processing of these materials, the mathematical modeling for performance and failure prediction of these materials, and the application of these models to the design of structural hardware with identification of research needs to improve this design process. A one-day workshop, to immediately follow the short course, is designed primarily for faculty of universities along with personnel of industrial educational units to discuss the content and pedagogical design in the course offerings 476 and 496.

### Development of Design Methodology Through Independent Studies

The initial application of brittle material design methods has been limited to the application of ceramics in the gas turbine engine. The faculty realizes the desirability of broadening the scope of these studies to include additional potential applications. In this direction the following two studies were undertaken during the report period.

#### 1. Space Shuttle Thermal Protection System

##### Faculty:

- R. J. H. Bollard, Coordinator, Aeronautics & Astronautics
- A. F. Emery, Mechanical Engineering
- B. J. Hartz, Civil Engineering
- A. S. Kobayashi, Mechanical Engineering
- W. J. Love, Mechanical Engineering
- A. D. Miller, Ceramic Engineering
- J. I. Mueller, Ceramic Engineering
- W. D. Scott, Ceramic Engineering
- R. Taggart, Mechanical Engineering
- O. J. Whittemore, Ceramic Engineering

**Staff:**

F. D. Gac, M.S., Ceramic Engineering  
R. D. Pfaff, B.S., Aeronautics & Astronautics  
A. M. Phister, B. S., Ceramic Engineering

**Graduate Students:**

T. R. Bieler, Ceramic Engineering  
D. W. Gilbert, Ceramic Engineering  
C. Georgiadis, Civil Engineering  
L. R. Chang, Aeronautics & Astronautics  
M. Kani, Aeronautics & Astronautics  
C. W. Newquist, Ceramic Engineering

**Undergraduate Students:**

B. A. Hassler, Ceramic Engineering  
E. S. Tosaya, Ceramic Engineering

The thermal protection system (TPS) of the Space Shuttle in the high temperature zones consists primarily of brittle tiles made from high purity silica ( $\text{SiO}_2$ ) fibers and typically measuring 6" x 6" x 2", attached to the surface of the contrastingly flexible aluminum vehicle through a low stiffness felt-like strain isolation pad (SIP). A study of the application to this TPS system of the design methodology in this program was undertaken to determine whether the approach would identify sources of problems the actual system was experiencing and to explore the potential of the methodology to the prediction of the system life-time under thermal and mechanical loading inputs typical of planned mission profiles.

The project has been divided into three interrelated sections, as follows:

- (1) Material Characterization--The objectives of this effort are to investigate and understand the structural characteristics of the strain isolation pad material and the influence of the various RTV bonding and RSI surface preparation techniques and the mechanical response under thermal and mechanical loadings.
- (2) Mathematical Modeling--The objectives of this section are to mathematically model (a) the load transfer mechanism at the SIP/RTV/Tile interface, (b) the nonlinear characteristics of the SIP and (c) the behavior of the TPS system as a typical installed tile, including thermal stress and stable crack growth.
- (3) Crack Initiation and Growth-Lifetime Prediction--The objective of this section is to establish crack initiation, growth and failure criteria which can be used in the life prediction of the SIP/RTV/Tile system.

Initial success has been achieved in all areas and work is continuing including coordination with other NASA and industry study teams involved. Separate, detailed reports on this phase of the program will be published as appropriate.

## 2. Britalus Engine Feasibility Study

### Faculty:

W. J. Love, Coordinator, Mechanical Engineering  
R. J. H. Bollard, Aeronautics and Astronautics  
A. F. Emery, Mechanical Engineering  
B. J. Hartz, Civil Engineering  
J. I. Mueller, Ceramic Engineering  
W. D. Scott, Ceramic Engineering  
R. Taggart, Mechanical Engineering

Combustion Research and Technology, Inc., a Seattle-based organization, has developed the design of a unique continuous combustion rotary piston engine. With funding from CR & TI, a study was made upon the feasibility of utilizing ceramic components in the current design in order to attain higher operating temperatures. The faculty team utilized the probabilistic design techniques developed by this program to access this problem. A report was submitted to the sponsor and will be used as case study in future course work.

## SUPPORTIVE RESEARCH

### Coordinating Committee

A. E. Gorum, Ceramic Engineering, Chairman (June 16 - September 15)  
J. I. Mueller, Acting Chairman (September 16 - December 15)  
A. F. Emery, Mechanical Engineering  
J. L. Bjorkstam, Electrical Engineering  
W. D. Scott, Ceramic Engineering  
R. G. Stang, Metallurgical Engineering

### Objectives

The goal of the research portion of this program is to keep abreast of the overall program in high performance structural ceramics in this country and elsewhere, and to do research in those areas that are deemed most appropriate whether it be related to materials or to structures. This should result in the University of Washington being a central focus for information and research in the area of high performance ceramics. The objective for the current year is to continue those programs initiated earlier and to continue developing interest among the University faculty in research areas deemed appropriate. One new program was initiated this year under funding by the Department of Army.

### Organization

Emphasis at the present time is on the structures and related materials programs in a ceramic turbine engine or other heat engines that would benefit from this type of material. The research program has been established so that all departments in the University that may contribute to the program are aware of the goals of the program and they participate in defining the individual areas of research that are to be undertaken. Those departments now involved are Physics, Aeronautics and Astronautics, Mechanical Engineering, Electrical Engineering, Civil Engineering, Metallurgical Engineering, and, of course, Ceramic Engineering.

The general planning of the program is carried out by the Ceramic Engineering faculty and coordinated by the above names. The overall research program is derived by analysis of proposals submitted from various departments of the University of Washington with emphasis being on the relationship of the proposed work to the needs defined in the area of structural ceramics.

### Progress

Projects are being supported at the present time. These will be detailed in the following section. Additional funding is being sought in the general area of high performance ceramics. When new funds are obtained, they will be used to support selective segments or extensions of the present program, and the present funds will be used to support additional programs. The progress on each project is given in the following reports.

RESEARCH ON HIGH PURITY MATERIALS ( $\text{Si}_3\text{N}_4$  and  $\text{SiC}$ )

Faculty Supervisor: A. E. Gorum, Research Professor and  
J. I. Mueller, Professor  
Ceramic Engineering

Graduate Assistant: Charles Newquist  
Research Assistant

Purpose

The purpose of this project is to:

1. Establish base line data (structure-property relationships).
2. Understand the role of additives, if they are really necessary, in various kinds of processing.
3. Evaluate the role of whiskers in reaction bonding silicon nitride (RBSN).

This will be accomplished most adequately by dividing the project into specific areas as follows:

1. Using course grained metallurgical grade silicon, the morphology of whisker growth, the structure of the whiskers, the effect of grain size and distribution on the amount and type of whiskers formed will be observed. Most importantly, the role of the whiskers in the final mechanical properties of reaction bonded  $\text{Si}_3\text{N}_4$  will hopefully be defined.

The properties and whisker morphology of whiskers obtained from metallurgical grade silicon will be compared with those grown from high purity silicon. This comparison would define to some extent the effect of additives.

2. Preparation and evaluation of high purity reaction bonded  $\text{Si}_3\text{N}_4$  to obtain base line data for high purity material.

Fine grained compacts of high purity silicon will be nitrided and the progress of the nitriding progress observed by ceramography. This amount and character of porosity as well as unreacted silicon will be measured and the mechanical properties of high purity reaction bonded  $\text{Si}_3\text{N}_4$  determined.

The optimum grain size and distribution will be postulated and specimens made to optimize the structure. This may well involve bi- or tri-modal distribution to optimize density as well as to control the reaction bonding characteristics.

### Relevance

By understanding the behavior of high purity materials prepared by different techniques, and the role of structural components such as whiskers, grain size, grain boundaries, etc., it should be possible to make materials that relate to specific applications whether it might be a heat exchanger tube or a turbine rotating part. It also allows tradeoffs as to properties, energy requirements for certain processes, etc.

### Objectives

The objective for the current year is to identify the role of iron as an additive to silicon as a sintering aid. This will be accomplished with assistance from other projects in the program.

### Progress

The Brew furnace is now operating on a routine basis and samples are being prepared for this program as well as others. A flow-through furnace for nitriding of special materials is in operation. This will allow wetting studies of additives as applied to sintering. The work on sintering, while starting on NASA funds, is being supported by another agency.

During the preparation of high purity silicon by leaching out impurities, it was very difficult to filter the fine material. It was decided to use a centrifuge to remove the solids and decant the liquid. The result was a dense green compact. This method appears to be quite satisfactory.

Leaching of high purity silica milled with iron balls does not appear to remove the iron contamination work reported by Professors Stern and Fain in the two projects immediately following, offer possible reasons for this.

## SURFACE CHARACTERIZATION OF SILICON NITRIDE

Faculty Supervisors: E. A. Stern, J. G. Dash, Professors, Physics  
S. M. Heald, Research Associate

Graduate Assistant: C. Bouldin, Research Assistant

Purpose

We propose to identify and characterize the surface properties of  $\text{Si}_3\text{N}_4$  that are important to the sintering processes by which this material is produced. This will be done by the coordinated application of two different probes of the surface properties: adsorption isotherm and extended x-ray absorption fine structure (EXAFS) measurements. The isotherms will be used to identify the population distribution of different types of adsorption sites characterized by their differing atom binding energies. The microscopic structure of these sites on an atomic scale can then be determined by the EXAFS measurements. Studies of samples produced under varying conditions should allow us to correlate the microscopic surface properties with sintering parameters.

Relevance

At present, detailed knowledge on an atomic scale of the microscopic physical processes involved in sintering is not available. The present work is aimed at developing such knowledge for one particular material,  $\text{Si}_3\text{N}_4$  but with the expectation that much of the same knowledge can be carried over to other materials. Once the physical basis of the sintering process is better understood, then this knowledge can be applied to optimizing the fabrication of technologically important products. In the case of  $\text{Si}_3\text{N}_4$ , these include components exposed to high temperature environments such as high performance gas turbines and combustion chamber. In the past, much of this optimization has been on a trial and error basis.

Objective

The first year of this project was devoted to construction and testing of necessary equipment, and the beginning of initial isotherm measurements. From the results of these initial measurements we were able to refine our equipment and prepare for making EXAFS measurements. EXAFS measurements began during this period, the beginning of the second year, on the better characterized system of Kr on graphite in order to fully develop the use of adsorbed Kr as a surface probe. Also, initial EXAFS measurements will begin on  $\text{Si}_3\text{N}_4$  as soon as suitable samples are available in the second or third year. Any further isotherm measurements suggested by the EXAFS results will also be made during the third year. The Fe-impurities in the  $\text{Si}_3\text{N}_4$  samples will be monitored by EXAFS to determine the change in their atomic and chemical environment during the sintering process.

Progress

In this reporting period we have continued our measurements of the Fe-impurities in  $\text{Si}_3\text{N}_4$  and have observed changes in the environment of these impurities at various stages in the processing. Since our previously reported measurements we have made improvements in our experimental

technique that have resulted in an order of magnitude improvement in signal to noise ratio. We have also made degree of disorder around the iron site.

The stages of the processing chosen for study were the starting Si powder, the final nitrided  $\text{Si}_3\text{N}_4$ , and the intermediate stage which involved the highest temperature - the sintered Si powder. The Fe environment of the Si powder is found to consist of oxygen neighbors with no evidence of other Fe as neighbors. In the intermediate stage after the Si powder has been sintered at  $1200^\circ\text{C}$  we see that the iron has moved into the bulk silicon and is surrounded by Si and other Fe atoms. In the final stage, after nitridation is completed, we find presumably either oxygen or nitrogen and by other Fe atoms. Analysis of our new measurements is continuing in order to make these conclusions more quantitative.

In addition to our EXAFS measurements we have completed construction of equipment for adsorption isotherms of Kr on  $\text{Si}_3\text{N}_4$ . Extensive isotherm measurements will begin in early 1980 along with the first EXAFS measurements of Kr on the  $\text{Si}_3\text{N}_4$  surface.



CHARACTERIZATION OF SILICON NITRIDE BY AUGER ELECTRON SPECTROSCOPY  
AND LOW-ENERGY ELECTRON DIFFRACTION

Faculty Supervisor: S. C. Fain, Jr., Associate Professor  
Physics

Graduate Assistant: A. G. Schrott  
Research Assistant

Purpose

To study the structure and composition of silicon nitride surfaces using low-energy electron diffraction (LEED) and Auger Electron spectroscopy.

Relevance

- A. Better physical models appropriate to describe bonding in silicon surface and bulk compounds might result from this research.
- B. Improvements in reaction bonding processing might result from a better characterization of the properties of  $\text{Si}_3\text{N}_4$  surface layers, and from studying the role of defects and impurities in nitridation.

Objectives

- A. Understand the process and products of nitridation of silicon single crystals in an ultra clean environment where LEED and Auger surface characterization can be used.
- B. Determine the effect of defects such as steps and of selected surface impurities such as iron on the nitridation of silicon single crystals.
- C. Assess the usefulness to reaction bonding process optimization of Auger electron spectroscopy analysis of surfaces obtained by fracture in ultra-high vacuum.

Progress

- A. Some results regarding the nitridation process and products for clean silicon (111) surfaces have been mentioned in previous status reports. In addition, a manuscript was presented at the New York American Vacuum Society Symposium and was accepted for publication. Further measurements are planned for other surfaces of silicon single crystals.
- B. Iron is known to play a significant role in the nitridation process by removing the silica film and by affecting the equilibrium between  $\alpha$  and  $\beta$  phases.<sup>1-3</sup> We were unable to locate any published LEED or Auger studies for the effects of iron on silicon single crystal surfaces or on the nitridation of otherwise clean crystal surfaces. Thus we designed a

method for depositing small amounts of ultrapure iron on the surface of otherwise clean silicon crystals. Our preliminary results for the Si (111) surface indicate that the iron goes into the bulk silicon well below 1000°C and thus it cannot be very important to nitridation in this context. The surface activity of iron will also be determined for other silicon single crystal surfaces.

The feasibility of studying nitridation on silicon single crystal surfaces with controlled step density is being investigated as a way to determine the effect of surface defects on the initial stages of nitridation.

- C. Two silicon nitride bars suitable for in-situ fracture and Auger studies were prepared from commercial RBSN material by F. Gac. The first measurements will be made after additional bars are obtained from RBSN prepared at the University of Washington from different starting powders.

#### Paper Presented

"Nitridation of Silicon (111): Auger and LEED Results," by J. F. DeLord (visiting scholar, Oct. 1978-July 1979), A. G. Schrott, and S. C. Fain, Jr., New York American Vacuum Society Symposium, Oct. 3, 1979 and accepted for publication in the Jan./Feb. 1980 issue of the Journal of Vacuum Science and Technology.

#### References

1. M. Mitomo, Journal of Materials Science 12, 273, (1977).
2. S. M. Boyer, A. J. Moulson, Journal of Materials Science 13, 1637 (1978).
3. D. Campos-Loriz et al., Journal of Materials Science 14, 2325 (1979).

## THE REACTION BONDING OF SILICON NITRIDE: PHYSICAL STRUCTURE RELATIONSHIPS

Faculty Supervisor: O. J. Whittemore, Professor  
Ceramic Engineering

Graduate Assistant: J. P. Chakraverty  
Research Assistant

Purpose

To study the nitriding of silicon to  $\text{Si}_3\text{N}_4$  by characterizing the pore size distribution, permeability, particle size distribution, appearance, porosity and density.

Relevance

Silicon nitride is being applied in dynamic applications requiring strength, refractoriness, and thermal shock resistance. The process of manufacturing silicon nitride by nitriding silicon compacts offers economic advantages since little dimensional change occurs. However, since the porosity, bulk and true densities, and phase change, much could be learned by characterizing the structure before and after nitriding. This study thus is another addition to our development of the science of pore morphology.

Objectives

To characterize the physical structure of silicon compacts and their subsequent nitrided state and to correlate with processing parameters as produced by other laboratories.

Progress

Permeabilities have been measured on sintered Si samples and their corresponding nitrided samples from Source 1, for which mercury porosimetry data were given in the last report. These are given in Table 1 in millidarcies together with surface areas calculated also from mercury intrusion, from integration of the particle size analyses assuming spheres, and from nitrogen adsorption (BET). Correlations of surface area by different methods is poor but within an order of magnitude.

Table 1. Permeability Data - First Set from Source 1

17

	<u>A<sub>1</sub>B<sub>1</sub>D<sub>1</sub></u>	<u>A<sub>1</sub>B<sub>1</sub>D<sub>2</sub></u>	<u>A<sub>1</sub>B<sub>2</sub>D<sub>1</sub></u>	<u>A<sub>1</sub>B<sub>3</sub>D<sub>1</sub></u>	<u>A<sub>3</sub>B<sub>5</sub>D<sub>1</sub></u>
Density,g/cc					
As-cast	1.72	1.74	1.67	1.75	-
Sintered	1.70	1.72	1.60	1.70	1.67
Nitrided	2.68	2.67	2.50	2.60	2.67
Permeability,mD					
Sintered	0.34	0.49	0.70	0.30	0.84
Nitrided	0.0027	0.0022	0.019	-	0.0032
Surface Area,m <sup>2</sup> /g					
As cast, BET	6.5	6.5	7.2	7.5	-
As cast, Hg intrusion	8.7	8.6	1.8	9.6	-
As cast, Particle size	1.8	1.8	1.9	1.9	-
Sintered, Permeability	2.0	2.6	2.9	4.2	2.5
Sintered, Hg intrusion	6.2	6.2	1.7	5.0	4.7
Nitrided, Permeability	7.4	6.8	3.7	-	8.5
Nitrided, Hg intrusion	6.0	5.4	3.5	4.5	5.2

A second set of samples was received from Source 1 and the data from mercury<sup>18</sup> intrusion are reported in Table 2. Not all pores were filled at 15000 psi (0.012  $\mu\text{m}$ ) as the cumulative penetration curve was still climbing.

Table 2. Physical Structure Data - Second Set from Source 1

	<u>Pore Fraction</u> <u>cc/cc</u>	<u>Surface Area</u> <u><math>\text{m}^2/\text{g}</math></u>	<u>Mid Pore Dia, <math>\mu\text{m}</math></u>	<u>Pore Fraction</u> <u>cc/cc</u>	<u>Surface Area</u> <u><math>\text{m}^2/\text{g}</math></u>	<u>Mid Pore Dia, <math>\mu\text{m}</math></u>
As Cast, $\text{Cr}_2\text{O}_3$ Additive	0.26	5.0	0.18			
	<u>Sintered in Vacuum</u>			<u>Nitrided After Sintering</u>		
2000°F. -1h	0.27	4.4	0.19	0.06	4.4	0.019
1900°F. -4h	0.26	3.9	0.20	0.06	4.2	0.019
1800°F. -4h	0.28	4.8	0.18	0.06	4.4	0.020
	<u>Sintered in Argon</u>			<u>Nitrided After Sintering</u>		
2000°F. -4h	0.27	4.1	0.19	0.05	4.2	-
1900°F. -1h	0.27	4.6	0.18	0.06	4.4	-
1800°F. -1h	0.28	5.0	0.18	0.06	4.3	-
<hr/>						
As Cast, $\text{Fe}_2\text{O}_3$ Additive	0.29	5.8	0.17			
	<u>Sintered in Vacuum</u>			<u>Nitrided After Sintering</u>		
1900°F. -1h	0.27	4.2	0.20	0.04	3.2	0.02
1800 -1h	0.27	4.6	0.19	0.03	2.6	0.02
	<u>Sintered in Argon</u>			<u>Nitrided After Sintering</u>		
2000°F. -4h	0.26	3.5	0.22	0.03	3.0	-
2000 -1h	0.26	3.8	0.20	0.03	2.9	-
1900 -4h	0.27	4.0	0.20	0.04	3.6	-
1800 -4h	0.27	4.2	0.19	0.03	3.1	-

A series of silicon and nitrided silicon samples were prepared at the University of Washington which had been sintered at various times at 1050 and 1200°C. The data of the 1050°C samples are presented in Table 3. These demonstrate pore growth during sintering, probably from a combination of surface diffusion and evaporation/condensation.

Table 3. Physical Structure Data of Silicon Compacts Sintered at 1050°C at Various Times and of Samples Nitrided After Sintering

	<u>1 hour</u>	<u>6 hours</u>	<u>12 hours</u>	<u>24 hours</u>
Density, g/cc				
Sintered	1.63	1.63	1.63	1.63
Nitrided	2.58	2.60	2.62	2.59
Pore Fraction, cc/cc				
Sintered	0.30	0.31	0.29	0.29
Nitrided	0.15	0.16	0.16	0.16
Surface Areas, m <sup>2</sup> /g				
Sintered	5.6	3.7	2.9	2.2
Nitrided	9.1	7.8	6.2	6.7
Mid Pore Dia., μm				
Sintered	0.18	0.27	0.30	0.42
Nitrided	0.06	0.08	0.10	0.11

Interpretation of the above data has been difficult, partly due to some discrepancies. Some of the samples of Table 2 have been tested for micropore volume by carbon tetrachloride adsorption and show presence of some porosity beyond that measured by mercury intrusion. More of these adsorption tests are planned together with surface area measurements by nitrogen adsorption and true specific gravities. With these additional data, Mr. Chakravorty plans to complete his Masters thesis by March 1980.

Faculty Supervisor: A. D. Miller, Associate Professor  
Ceramic Engineering

Staff Assistant: F. D. Gac, Research Engineer  
Ceramic Engineering

### Purpose

The purpose of this project is to assess the relationship between microstructural features and failure behavior for brittle materials of interest.

### Relevance

Precise understanding of the microstructural features accompanying failure would be very useful for developing structural ceramics whose fracture sensitivity is reduced. Such information would be important not only to the materials community, but also other disciplines participating in design with brittle materials, such as aeronautical, civil, and mechanical engineering.

### Objectives

- A. Develop a facility and expertise necessary for examination and interpretation of failed brittle material test specimens. (Included in this is the careful review of work by other investigators, and verification of our work by these investigators.)
- B. Examine and determine the relationship between microstructural features and failure behavior for test specimens generated elsewhere and at this University, particularly those from projects under NASA sponsorship.
- C. Establish a method for reporting fractography results, and compile these results into a single or multi-volume publication on fractography of brittle materials.

### Progress

Fulfillment of all three objectives is well underway. Materials examined thus far include reaction bonded  $\text{Si}_3\text{N}_4$  (RBSN) modulus of rupture (MOR) bars, supplied by the AiResearch Casting Co. The first batch of samples were formed by injection molding, and the characteristic fracture origin appeared to be laminar flaws resulting from the molding operation. A second batch of bars, formed by slip casting, are currently under evaluation.

In conjunction with the Brittle Materials Design Program dealing with the A. C. Spark Plug Exhaust Gas Sensor, substantial work was performed in examining the fractured zirconia sensors, MOR bars, and biaxial test disks. The fractography results confirmed the computer stress analysis predictions, and also assisted in discovering the flaws present on the interior surface of the sensor, which were responsible for failure sensors considered satisfactory by dye penetrant test on the exterior of the sensor.

Work is also underway in evaluating the fracture behavior of the thermal protection system for the NASA Space Shuttle. An important portion of this work also includes thorough microscopic characterization of the as-received components prior to testing to failure.

Finally, work is also continuing in examination of RBSN specimens fabricated at this University, with the objective of optimizing the quality of the components produced. We are now nearly achieving the density and strengths reported by industry.



Faculty Supervisor: Robert G. Stang, Assistant Professor  
Metallurgical Engineering

Graduate Assistant: Raymond D. Mar  
Research Assistant

### Purpose

Study the high temperature creep behavior of commercially manufactured  $\text{Si}_3\text{N}_4$  and to compare the creep behavior of the commercial material with  $\text{Si}_3\text{N}_4$  manufactured by the processing group at the University of Washington. This work will focus on attempts to measure the strain time behavior of both types of  $\text{Si}_3\text{N}_4$  as a function of stress and temperature. A model, based on observed high temperature deformation mechanisms will be developed to describe the behavior as a function of stress and temperature.

### Relevance

The microscopic mechanisms which control high temperature plastic deformation must be determined to identify the best method for strengthening materials. For materials used in creep applications, this is often done by determining the stress and temperature dependencies. This data is used to formulate a model which describes the strain rate as a function of stress and temperature. The effects of impurity additions and microconstituents on the parameters in models of this type can be used to optimize and/or predict the deformation behavior of the material under study for different stress and temperature conditions.

### Objectives

- A. Design and construction of a compression creep machine capable of operating in air at temperatures up to  $1450^\circ\text{C}$  and at stresses up to 552 MPa (80,000 psi).
- B. Comparison of strain-time data, as a function of stress and temperature, generated using our equipment with data available in the literature. The purpose of this phase of the program is to check the apparatus and experimental procedure.
- C. Investigate the creep behavior of material produced by the processing group as a function of stress and temperature.
- D. Attempt to identify the rate controlling process for deformation in the material under study so that it might be strengthened.

### Progress

Premature fracture of the sample and/or platten material has been a major problem. New platens manufactured from  $\text{Si}_3\text{N}_4$  have been produced. In addition, a supply of SiC has been ordered for use as platten material.

It is possible that the cracking of the samples and/or plattens is caused by overloading during the initial heating cycle due to ram misalignment. Therefore, a load cell was recently purchased to monitor the sample load during the initial heating cycle to determine if this is a problem. The creep machine is currently being modified to incorporate the load cell in the load train. Finally, considerably more attention is being paid to specimen preparation.

Faculty Supervisor: John L. Bjorkstam, Professor  
Electrical Engineering

Graduate Assistant: Davene Eyres  
Research Assistant

### Purpose

The purpose of this investigation is to develop an opto-acoustic technique for detecting small ( $\lesssim 20\mu\text{m}$ ) subsurface (and surface) flaws in materials with high acoustic attenuation. In its final form the research has two major thrusts: (1) the generation of acoustical pulses with high energy density in the spectral range corresponding to wavelengths  $\lesssim 20\mu\text{m}$  (frequencies  $\gtrsim 200$  MHz), and (2) the detecting of acoustical energy scattered from flaws using a method which would be convenient in a production format.

### Relevance

This research should lead to improved methods for non-destructive-evaluation (NDE) of structural and electronic ceramics. The technique will have particular applicability to location of small inhomogeneities, of acoustic impedance different from the sample material, in materials with appreciable acoustic attenuation and normally smooth surfaces.

The practical engineering uses of lasers and acoustical techniques are closely related to several other projects underway within the electrical engineering department, thus providing a mutual strengthening of these projects. In addition, the interdisciplinary nature of the investigation has encouraged valuable professional interaction outside the EE department.

### Objectives

The primary objectives are, (1) to generate short high intensity, acoustic pulses using differential thermal expansion at a material surface which absorbs the output of a pico second laser pulse train, and (2) to detect and quantify the energy so generated in the frequency range  $\gtrsim 200$  MHz. While this method of high intensity acoustic pulse generation has been demonstrated in other laboratories, there is no reliable experimental data on opto-acoustic conversion efficiency. Thus our initial investigations have been directed toward a broad survey of formats for high conversion efficiency and sensitive, calibrated, detection methods.

### Progress

During the last six months the pico second pulse laser has at last been back in operational status. Initial attempts to detect the acoustical energy using strictly optical probing techniques have proven unsuccessful due to unwanted light scattering effects in the preliminary optical system.

The optical system is being improved and at the same time we are fabricating interdigital-finger surface-acoustic-wave (SAW) transducers. The SAW devices should have a sensitivity ~ 1-2 orders of magnitude greater than optical detection systems for the required system band widths. They have the disadvantage of requiring mechanical coupling to the sample but this can be accomplished through a liquid layer.

#### Correlative Research

As a result of the relationship between our work and acoustical microscopy the faculty supervisor on this project was invited to participate in a Workshop on Research in Otolaryngology at the Battelle Northwest Seminar and Study Center, 15 December 1979. The workshop was sponsored by the University of Washington Center for Biomedical Sciences and the Department of Otolaryngology.

#### Paper Presented

J. L. Bjorkstam and Davene Eyres, "Opto-Acoustic Generation of Ultrasonic Energy for Nondestructive Evaluation," presented at the American Ceramic Society 32nd Pacific Coast Regional Meeting; Seattle, Washington, 24-26 October 1979.

RELIABILITY PREDICTION OF THE STRENGTH OF CERAMIC MATERIALS  
UNDER COMPLEX STRESS STATES

Faculty Supervisor: B. J. Hartz, Professor  
Civil Engineering

Graduate Assistant: Osamu Inoue  
Research Assistant

Purpose

To develop the methodology and data necessary for the prediction of the statistical distribution of the strength of ceramic structural components under complex stress states.

Relevance

This project requires the utilization and further development of extremely important concepts of materials characterization, particularly under complex stress states, Weibull statistical representation of strength of brittle materials and refined stress analysis via finite element method. A particularly important aspect of this project is the large number of prototypical structural specimens being tested for the determination of the statistical distribution of the structural strength. This project is relevant both to the development of an academic program to teach design for brittle materials and to the needs of industry currently utilizing these design concepts for brittle materials but without the large statistical verification being undertaken in this project.

Objectives

The objectives of the current year are the implementation of a three dimensional FEM code for the analysis of the complex stress states on the notched alumina substrates being used as prototypical structures, the accumulation of statistical strength data on these specimens, and determination of the materials property of the alumina materials in these substrates.

Progress

Testing program has been completed including testing of the uniform and 2-d notched specimens obtained from slicing of the notched substrates. Data from 3 and 4 point MOR tests of the uniform specimens has been analyzed and basic Weibull parameters for the materials obtained. The Weibull parameters obtained from these tests were used in the Finite Element Analysis of 3 and 4 point loaded 2-dimensional beams obtained from slicing the substrates to predict the

Probability of failure curves for 2-dimensional notched specimens for comparison with the measured values. It was found that a very fine FEM mesh was needed in the vicinity of the notch in order to get a good comparison between calculated and measured values. A careful analysis of the relationship of the stress analysis performed and prediction of failure has been made.

It has not been possible with the limited effort on this project to complete the 3-dimensional Finite Element Analysis. The careful analysis and satisfactory results for the 2-d notched specimens has been gratifying.

#### Paper Presented

"Reliability Prediction of the Strength of Ceramic Materials Under Complex Stress States" by B. J. Hartz and Osamu Inoue, Pacific Regional Meeting for 1979, American Ceramic Society, Oct. 24-26, 1979, Seattle, Washington.

Faculty Supervisors: Albert S. Kobayashi, Professor  
Mechanical Engineering

Ashley F. Emery, Professor  
Mechanical Engineering

Graduate Assistant: Been Hing Benjamin Liaw  
Research Assistant

### Purpose

Since impact damage and erosion of structural ceramics are caused by dynamic loadings, it should be possible to construct phenomenological models of the above based on dynamic fracture mechanics. The purpose of this project is to explore such feasibilities with the hopeful goal of establishing impact damage and erosion models of structural ceramics.

### Relevance

Dynamic fracture mechanics is in its development stage and this impact damage and erosion studies based on dynamic fracture mechanics have not been unexplored. This project is therefore pushing the state-of-art in fracture mechanics.

Studies on dynamic fracture characterization of structural ceramics is an interdisciplinary undertaking in fracture mechanics and ceramic material research.

### Objectives

The first and second year objective is the dynamic fracture characterization of structural ceramics at room through elevated temperatures. The third objective is the development of a dynamic fracture model of impact damage. The fourth and fifth year objective is the development of an erosion damage model based on the above.

### Progress

Accomplishment: Work for this project was not undertaken until October 1, 1979. The results reported are thus for the period of October 1 through December 15, 1979. A dynamic fracture mechanic assessment of the impact fracture experiments by D. L. Johnson, Northwestern University is being conducted using dynamic elasticity and dynamic finite element analysis. Preliminary results show that the indicated low dynamic fracture toughness,  $K_{ID}$ , at elevated temperatures may be incorrect as suspected.

Problem areas: Lack of specific details on Johnson's experiments required estimates on some test fixture dimensions and materials for the above study. Since the purpose is to demonstrate the influence of dynamic fracture mechanics and not to correct Johnson's results, the missing information is not vital for this preliminary study.

Progress toward current year objective: Despite the 3-1/2 month delay in initiating work on this project, it is hoped that dynamic fracture characterization of typical structural ceramic, alumina, at room temperature will be underway by June 15, 1980. The expected progress will be governed to some extent on receiving NASA approval to purchase the TTI Fractomat for crack measurement in dynamic fracture specimens.

#### Paper Presented

"Fracture Toughness of Alumina At Room Temperature to 1600°C" by A. S. Kobayashi, A. F. Emery, A. E. Gorum and T. Basu, Pacific Regional Meeting for 1979, American Ceramic Society, October 23-26, Seattle, Washington.

#### Paper Published

"Fracture Toughness of Alumina at Room Temperature to 1600°C" by A. S. Kobayashi, A. F. Emery, A. E. Gorum and T. Basu, Proceedings of the Third International Conference on Mechanical Behavior of Materials (edited by K. J. Miller and R. F. Smith), Pergamon Press, 1979, Vol. 3, pp. 3-10.



Faculty Supervisors: Ashley F. Emery, Professor  
Mechanical Engineering

Albert S. Kobayashi, Professor  
Mechanical Engineering

Graduate Assistant: Tom Bieler  
Research Assistant

### Purpose

The applicability of ceramic materials in hostile environments will depend strongly upon the thermal sensitivity of the material and its resistance to thermal shock. The purpose of this project is to design thermal shock sensitivity tests for characterization.

### Relevance

Thermal sensitivity characterization is poorly done at the present time. This project will develop procedures for good industrial decisions as to the acceptability of materials for different conditions.

### Objectives

The objective is to analytically design realistic and reproducible thermal shock tests through analytical studies of the temperatures and thermal stresses which exist in several different test specimen geometries and thermal boundary conditions. Such analyses will permit us to choose among the alternatives, that test protocol which will most accurately define the thermal shock resistance of typical brittle ceramic materials.

### Progress

- A. A preliminary analysis of the thermal spot test has been carried out and the results reported at the Pacific Regional Meeting. This analysis determined the basic characteristics of the stresses in an uncracked specimen and was used to establish typical dimensions and testing times which would yield good fracture testing.
- B. The accurate assessment of thermal stresses depends upon an ability to predict temperatures. We have developed a highly versatile thermal analyzer, which appears to be one of the best currently available in the United States. This analyzer is undergoing final testing and should be complete by January 15, 1980.
- C. We have also developed plane and axisymmetric stress analyzers to be coupled with the thermal analyzers. The complete package is expected to be available by the end of winter quarter, and will be used in the final analysis of the proposed tests.

Paper Presented

"Analysis of the Spot Thermal Shock Ceramics Test" by A. F. Emery, A. S. Kobayashi and T. R. Bieler, Pacific Regional Meeting for 1979, American Ceramic Society, October 23-26, 1979, Seattle, Washington.

## EVALUATION OF TESTS FOR MECHANICAL PROPERTIES OF CERAMIC MATERIALS

Faculty Supervisor: R. J. H. Bollard, Professor  
Aeronautics and Astronautics

Graduate Assistants: Ms. H. Faou  
Research Assistant

Liang-Ruey Chang  
Research Assistant

### Purpose

To accurately assess the stress state under given load in specimens commonly used in brittle material characterization and failure tests and thereby assess the errors inherent in such tests and in the use of simple model approximations to the actual stress states. It is intended to extend this assessment to candidate tests not commonly used and to develop error assessments in the correlation of failure results between tests using different specimens, tests using specimens of the same geometry but different size, and, tests of different complexity in the stress field such as between uniaxial and multiaxial states.

### Relevance

Since theoretical behavior and failure predictions are based on stress functions and predicted stresses at observed failure, it is apparent that the accuracy of such predictions is directly related to the accuracy with which the stresses can be predicted. For example, in isotropic, homogeneous elastic materials the calculation of stresses and consequent behavior is dependent on the material properties  $E$  and  $\nu$  for isothermal conditions and the prediction of failure is based on the theoretical stress value at observed failure in a given test. Since the observed failure of brittle material in a given test usually occurs with a large scatter in values of failure load a statistical model of failure must be used. The most common such model is that due to Weibull and introduces a further material property,  $\sigma_0$ , and the Weibull parameter  $m$ , which is a measure of the scatter in the observed load at failure. This model is often refined by the additional parameter  $\sigma_u$ , that stress level below which failure is not observed to occur in a particular test.

In the prediction of failure it is particularly important to know the possible error occurring in the calculated values of  $\sigma_0$ ,  $\sigma_u$  and the actual stress state for in the Weibull model differences of ratios of these quantities are raised to the power of  $m$  and errors are thereby magnified. Furthermore, the accuracy of correlations in failure prediction between tests using specimens of the same geometry but different size, specimens of different geometry, or specimens in which the stress states are different, will be dependent on the accuracy with which the actual stress at failure is predicted. In complicated geometries and loading states these correlations require the knowledge of the stress field throughout the volume of the specimen which fact places further demand on the assessment of error in the derived stress as compared to those actually occurring.

### Objectives

The objectives for the first year and a half have been met by completion of assessments in beam bending MOR tests.

The objectives for the second year, already partially achieved, include the error assessment in MOR tests of different geometries but simple stress fields, including biaxial fields, and error assessments in correlations of predicted failure between different tests.

The continuing objectives are to develop an experimental program to support the theoretical predictions of failure in complex stress fields from simple stress state data.

### Progress

The error analysis of MOR data for simple 3 and 4 point bending has been completed using a Finite Element computer model to more accurately assess the tensile stress at failure. The results were presented at the AMMRC conference at Rosario, Washington, in July and will appear in the paper by F. I. Baratta titled Flexure Test Method T101 in the proceedings of that meeting. This paper is essentially a proposal to standardize the specimens used and the procedures in beam MOR testing. The results of this particular study appear as an appendix and are fully supportive of the expounded need for a standard. The results had the additional effect of correcting some erroneous results appearing in early editions of the paper and arising from improper superposition of simple analytical results for different geometries and loadings. The more accurate FEM analyses point out dramatically the large errors accompanying beam testing where the support and loading systems are not symmetrically arrayed and especially so in the case of the loading head being essentially rigidly held in the loading frame.

The results of error assessment in the correlation between failure predictions in MOR specimens of different size, the so-called volume effect, were completed in late summer and reported at the October meeting of the Pacific Coast Regional Meeting of the American Ceramic Society. This correlation study is currently being applied to similar geometries but different loading states.

### Paper Presented

"Error Assessment in Characterization Testing by Brittle Materials," by R. J. H. Bollard and H. Faou, Pacific Regional Meeting for 1979, American Ceramic Society, October 23-26, 1979, Seattle, Washington.

Participating Faculty

J. L. Bjorstam, Professor of Electrical Engineering  
R. J. H. Bollard, Professor of Aeronautics & Astronautics, Design  
Methodology Coordinator  
J. G. Dash, Professor of Physics  
A. F. Emery, Professor of Mechanical Engineering  
S. C. Fain, Jr., Associate Professor of Physics  
A. E. Gorum, Affiliate Professor  
B. J. Hartz, Professor of Civil Engineering  
S. M. Heald, Research Associate, Physics  
A. S. Kobayashi, Professor of Mechanical Engineering  
W. J. Love, Professor of Mechanical Engineering  
A. D. Miller, Associate Professor of Ceramic Engineering  
J. I. Mueller, Professor of Ceramic Engineering, Principal Investigator  
R. G. Stang, Assistant Professor of Metallurgical Engineering  
E. A. Stern, Professor of Physics  
W. D. Scott, Professor of Ceramic Engineering  
O. J. Whittemore, Professor of Ceramic Engineering

Staff

L. Blume, Secretary  
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C. Mitton, Secretary (part-time)  
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Research Assistants

T. K. Bieler, Ceramic Engineering  
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J. P. Chakraverty, Ceramic Engineering  
L. R. Chang, Aeronautics & Astronautics  
D. T. Evers, Electrical Engineering  
H. L. Faou, Aeronautics & Astronautics  
C. Georgiadis, Civil Engineering  
D. W. Gilbert, Ceramic Engineering  
M. Kani, Aeronautics & Astronautics  
O. Inoue, Civil Engineering  
B. H. B. Liaw, Mechanical Engineering  
R. D. Mar, Metallurgical Engineering  
C. W. Newquist, Ceramic Engineering  
A. G. Schrott, Physics

APPENDIX II  
CERAMIC MATERIALS RESEARCH BOARD

- J. I. Mueller, Professor of Ceramic Engineering  
Principal Investigator & Chairman
- B. W. Mar, Professor of Civil Engineering  
Associate Dean, College of Engineering
- J. G. Dash, Professor of Physics  
Representing the Dean, College of Arts & Sciences
- D. G. Dow, Professor of Electrical Engineering
- A. S. Kobayashi, Professor of Mechanical Engineering
- W. D. Scott, Professor of Ceramic Engineering
- T. G. Stoebe, Professor of Metallurgical Engineering

## American Ceramic Society 32nd Pacific Coast Regional Meeting

Washington Plaza Hotel, Seattle, Wash.  
October 24-26

### PROGRAM

#### INTERDISCIPLINARY SYMPOSIUM ON STRUCTURAL DESIGN WITH CERAMIC MATERIALS

Cosponsored by NICE and the local sections and chapters of AIAA, ASCE, ASME, SAE, SAMPE, and ADPA

**Wednesday, October 24**

#### Concord Room

##### "Interdisciplinary Activities"

Session Chairman: JAMES I. MUELLER, University of Washington, Seattle

**9:00 a.m. Welcoming Remarks by WILLIAM H. PAYNE, president, National Institute of Ceramic Engineers**

**9:05 a.m. Desirability of Intersociety Cooperation (1-SD-79P)**

MORTON KUSHNER, Boeing Aerospace Co., Seattle, Wash.

It has been recognized for years that the multiplicity of societies (at least 35 in number) with interest in materials science and engineering and related disciplines inevitably fosters duplication and effort, with resultant inefficient use of critical manpower and money. Better coordination of interrelated and interdisciplinary activities is needed to minimize fragmentation and dissipation of human resources.

**9:35 a.m. Failure Criteria in Designing Ceramic Structures (2-SD-79P)**

W. H. DUCKWORTH\* and A. R. ROSENFELD, Battelle, Columbus Labs, Columbus, Ohio

The designing of ceramic structures using an analytical approach imposes requirements for quantitative treatments of the size, time, and stress-state dependencies of failure stress. Available theories and relations to account for each effect and the conditions that must be met for their correct use are discussed.

**10:05 a.m. Need for Interdisciplinary Communication in Design and Development of Engineering Ceramics (3-SD-79P)**

A. F. McLEAN, Ford Motor Co., Dearborn, Mich.

Good communication between disciplines creates a basis for continual interchange and feedback, as well as a spirit of teamwork and enthusiasm necessary to foster success in this relatively new technological area. A case history is used to describe such interdisciplinary communication.

**10:30 a.m. Structural Design with Ceramic Materials (4-SD-79P)**

ANTHONY PALUSZNY, Ford Motor Co., Dearborn, Mich.

**11:00 a.m. Applying Brittle Design Theory to the Manufacture of Ceramic Components (5-SD-79P)**

M. SRINIVASAN\* and G. W. WEBER, Carborundum Co., Niagara Falls, N.Y.

The application of brittle material design theory to the processing and fabrication of actual components requires test data representative of component properties. The use of these results in the process development of sintered  $\alpha$ -SiC vehicular components is described.

**11:30 a.m. Interdisciplinary Studies Involving Ceramic Structural Materials (6-SD-79P)**

**\*\* J. I. MUELLER, R. J. H. BOLLARD,\* and R. TAGGART, University of Washington, Seattle**

A unique program of interdisciplinary studies on design methodology involved in the use of high performance ceramics in structural applications is discussed. This program has been supported at the University of Washington through a grant from NASA.

#### Concord Room

##### "Properties and Testing"

Session Chairman: WILLARD E. HAUTH III, Los Alamos Scientific Lab, Los Alamos, N.M.

**2:00 p.m. Ceramics for an Adiabatic Turbo Compound Engine (7-SD-79P)**

R. KAMO, Cummins Engine Co., Inc., Columbus, Ind.

The development of ceramic components for use in an adiabatic turbo compound diesel engine and the operational advantages derived are discussed.

**2:30 p.m. Processing and Structure of Low Sintering RBSN (8-SD-79P)**

F. H. SIMPSON and JURIS VERZEMNIEKS,\* Boeing Aerospace Co., Seattle, Wash.

The reaction paths in the RBSN process and their effects on the structure and properties of a Boeing-developed silicon nitride reduce material are discussed.

**3:00 p.m. Influence of Presintering of Silicon on the Final Properties of RBSN (9-SD-79P)**

**\*\* J. T. EWANICH and A. E. GORUM,\* University of Washington, Seattle**

Presintering of silicon prior to nitriding allows machining with carbide tools for fine finishing, and the dimensions will remain the same through final nitriding. The effect of this sintering on the final structure and properties of the reaction-bonded  $\text{Si}_3\text{N}_4$  is discussed.

**3:30 p.m. Nitrogen-Containing Crystal Structures (10-SD-79P)**

P. E. D. MORGAN, Rockwell International, Thousand Oaks, Calif.

Known nitrogen-containing crystal structures obey the normal rules of crystal chemistry. These rules are used to predict new types, which are currently being synthesized, by computer analysis of known structures.

**4:00 p.m. Error Analysis in Characterization and Failure Testing (11-SD-79P)**

**\*\* H. L. FAOU and R. J. H. BOLLARD, University of Washington, Seattle**

Finite element techniques were employed to assess potential errors in the experimental evaluation of failure states in simple stress fields and in the correlation of results between test specimens of different size.

**4:30 p.m. Reliability Predictions of the Strength of Ceramic Materials under Complex Stress States (12-SD-79P)**

**\*\* B. J. HARTZ\* and O. INOUE, University of Washington, Seattle**

A large number of notched alumina substrates were tested in MOR-type tests with varying test parameters. Predictions of the statistical distributions of strength for the different loading conditions were obtained using Weibull parameters determined for the material and various stress analysis approximations. These are compared with the measured strength distributions.

**Thursday, October 25**

#### Concord Room

##### "Fracture Mechanics: I"

Session Chairman: ALBERT S. KOBAYASHI, University of Washington, Seattle

**9:00 a.m. Transformation Toughening in Ceramics (13-SD-79P)**

N. BURLINGAME, M. DRORY, A. G. EVANS,\* and W. M. KRIVEN, University of California, Berkeley

The toughening induced by martensitic transformations in ceramics is

discussed. The role of the chemical free energy is related to chemical composition and temperature effects. Calculations of the toughening are described and used to illustrate the toughening trends.

**9:30 a.m. High Temperature Failure Mechanisms in Ceramics (14-SD-79P)**

W. BLUMENTHAL, A. G. EVANS,\* and J. PORTER, University of California, Berkeley

High temperature failure in ceramics occurs by the nucleation, growth, and coalescence of cavities through diffusive or viscous flow processes. Models of the failure process are described, indicating specific roles of grain size, chemical composition, diffusivity, etc. Deformation and fracture data provide a basis for correlation with the cavitation models.

**10:00 a.m. Effect of Electric Fields on Slow Crack Growth in Glass: Theory of the Transient Effects (15-SD-79P)**

S. D. BROWN\* and M. K. FERBER, University of Illinois, Urbana

The effects of electric fields on slow crack growth in glass are reviewed briefly. Theoretically, the effects are treated as electrostrictive phenomena from the standpoint of the multibarrier kinetics treatment of slow crack growth. Emphasis is placed on the nature of transient behavior.

**10:30 a.m. Strength and Fracture Toughness of Aluminum Silicate Materials (16-SD-79P)**

P. C. DOKKO and J. A. PASK,\* University of California, Berkeley

Bending strength, compressive strength, and fracture toughness were determined for single-phase mullite and multiphase aluminum silicate materials with mullite as a major phase at room temperature and high temperatures. Retained strengths after thermal shock were measured. Correlations between mechanical properties and microstructure were determined.

**11:00 a.m. Analysis of the Spot Thermal Shock Ceramics Test (17-SD-79P)**

**\*\*** A. F. EMERY,\* A. S. KOBAYASHI, and T. R. BIELER, University of Washington, Seattle

A numerical analysis was made to evaluate the thermal stresses generated in a disk of radius  $R$  by the heating of a surface spot of radius  $r$ . Transient stress intensity factors were calculated for several values of  $r/R$  and different crack orientation to determine the value of the specimen in ranking the thermal shock toughness of ceramics.

**11:30 a.m. Thermal Stress Studies of High Performance SiC (18-SD-79P)**

K. T. FABER,\* Carborundum Co., Niagara Falls, N.Y., and A. G. EVANS, University of California, Berkeley

Continuing studies on the development of a thermal stress test designed to subject materials to rapid temperature changes where  $\beta$  is  $< 20$  are described. Empirical results determined for two high performance silicon carbides are discussed.

**Concord Room**

**"Fracture Mechanics: II"**

Session Chairman: JOHN S. NADEAU, University of British Columbia, Vancouver, B.C., Canada

**2:00 p.m. Fracture Toughness of Alumina at Room Temperature to 1600°C (19-SD-79P)**

**\*\*** A. S. KOBAYASHI,\* A. F. EMERY, A. E. GORUM, and T. BASU, University of Washington, Seattle

A wedge-loaded double cantilever beam specimen was developed for fracture toughness testing and subcritical crack growth measurements at high temperature. Fourteen fracture specimens were tested at room tempera-

ture and from 600° to 1600°C in vacuum. The resultant fracture toughness was a relatively constant value of 5.2 MPa $\sqrt{m}$ .

**2:30 p.m. Fracture Mechanism by Mechanical Stresses in Ceramic Materials for Building (20-SD-79)**

CARLO PALMONARI\* and GABRIELE GAVIOLI, Centro Ceramico, Bologna, Italy

To evaluate the fracture behavior of building ceramics, especially floor and wall tiles, maximum principal stresses in fixed ceramic and whiteware tiles, as well as elastic moduli, were determined. Correlations of elastic modulus and bending resistance with primary physical properties of fired and glazed tiles were found.

**3:00 p.m. Proof Testing of Ceramics: Effect of Multiregion Crack Growth (21-SD-79P)**

S. W. WIEDERHORN,\* E. R. FULLER, and S. W. FREIMAN, NBS, Washington, D.C.

Theoretical estimates of strength distributions in chemically active environments are presented. Results are compared with experimental data obtained on soda-lime silicate glass. The importance of Region II crack growth to the shape of the strength distribution curve is discussed.

**3:30 p.m. PANEL DISCUSSION—"Fracture Properties of High Temperatures"**

Panelists: A. G. EVANS, moderator, E. F. EMERY, A. S. KOBAYASHI, J. S. NADEAU, and S. M. WEIDERHORN.

**Friday, October 26**

**Concord Room**

**"Reliability and Nondestructive Evaluation"**

Session Chairman: FRANK D. GAC, University of Washington, Seattle

**9:00 a.m. Reliability of High Performance Ceramics (22-SD-79P)**

S. A. BORTZ, IIT Research Institute, Chicago

The measurement of reliability of structural ceramics combining mechanical strength, fracture mechanics, NDE, and design, is discussed.

**9:30 a.m. Nondestructive Evaluation of Gas Turbine Ceramics—A Status Report (23-SD-79P)**

J. J. SCHULDIES, AiResearch Manufacturing Co., Phoenix, Ariz.

An overview of the state of the art of NDE as applied to gas turbine components is presented. Various conventional and emerging NDE techniques were investigated to assess their potential for detecting flaws in HPSN and RBSN. Detect/NDE standards were used to rank the various methods in terms of detection sensitivity.

**10:00 a.m. Opto-Acoustic Generation of Ultrasonic Energy for Non-destructive Evaluation (24-SD-79P)**

**\*\*** J. L. BJORKSTAM and DAVENE EYRES, University of Washington, Seattle

The absorption of picosecond optical laser pulses at a sample surface may be used to generate intense pressure acoustical waves. The advantages of this method are discussed with respect to controlling the resulting ultrasonic frequency spectrum, acoustical aperture, and beam scan for NDE purposes.

**10:30 a.m. Microwave Nondestructive Evaluation of Ceramics (25-SD-79P)**

A. M. BAHR, Stanford Research Institute, Palo Alto, Calif.

Microwave frequency electromagnetic waves can penetrate some structural ceramics such as  $\text{Si}_3\text{N}_4$ ; these waves are thus potentially useful for detecting and characterizing internal flaws in such materials. The results of transmission and backscattering measurements are presented, and various factors related to the practical utility of microwave NDE are discussed.

**\*\*Denotes Paper Presented**



## APPENDIX IV

CERAMIC STRUCTURAL MATERIALS REVIEW

July 9, 1979

## List of Attendees

Marc Adams	Jet Propulsion Laboratory
Robert Baker	Ford Motor Company
Joe Battenberg	Eaton Corporation
Morris Berg	AC Sparkplug Division, GM Corporation
Seymour Bortz	Illinois Institute of Technology Res. Inst.
John Brennan	United Technology
W. Bunk	DFVLR, Cologne, W. Germany
Dave Carruthers	AiResearch Manufacturing Company
Winston Duckworth	Battelle Columbus Laboratories
Jim Gangler	NASA Headquarters
Chet Hinman	Westinghouse Hanford
John Hurt	Army Research Office
Bob Jones	Hughes Aircraft
Roy Kamo	Cummins Engine Company
Bob Katz	AMMRC
Jim Keski	Tektronix, Inc.
Ed Kraft	Carborundum Company
Gerald Kravik	Pacific Grinding Wheel
Wurth Krieger	Consultant
S. O. Kronogard	United Turbine, Sweden
J. G. Lanning	Corning Glass Works
Hyman Leggett	Hughes Aircraft
Howard Larson	NASA - Ames Research Center
Martin Leopold	Jet Propulsion Laboratory
L. Malmrup	United Turbine, Sweden
H. Mizuhara	GTE-Wesgo
Tony Paluszny	Ford Motor Company
Dick Phoenix	Carborundum Company
Ken Porter	Boeing Company
Bert Probst	NASA - Lewis Research Center
Roy Rice	Naval Research Laboratory
J. E. Ritter	University of Massachusetts
Mike Rorabaugh	AiResearch Casting Corporation
Jack Rubin	Kyocera International
Vic Saffire	General Electric Company
Karsten Styhr	AiResearch Casting Corporation
Juris Verzemnieks	Boeing Company
Gerald Weaver	Norton Company
Sheldon Wiederhorn	National Bureau of Standards
Ed Wright	AMMRC

APPENDIX V  
ENROLLMENTS - 1979

<u>Department</u>	<u>Graduate</u>	<u>Total</u>
<u>536</u> - Summer		
Ceramic Engineering	6	6
Civil Engineering	2	2
	—	—
	8	8
<u>536</u> - Autumn	2	2

Students Enrolled In Design Methodology Courses

Ceramic Engineering

Basu, T. K.	(Grad)	536
Bieler, T. R.	(Grad)	536
Chakraverty, J.	(Grad)	536
Ewanich, J. T.	(Grad)	536
Gac, F. D.	(Grad)	536
Hannan, J. K.	(Grad)	536
McLaren, D.	(Grad)	536

Civil Engineering

Georgiadis, C.	(Grad)	536
Inoue, O.	(Grad)	536

APPENDIX VI  
DESIGN WITH BRITTLE MATERIALS  
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Unit 3.	Theory of Elasticity	R. J. H. Bollard
Unit 4.	Mechanical Characterization	R. J. H. Bollard, R. Taggart
Unit 5.	Effect of Ceramic Microstructure on Mechanical Properties	W. D. Scott
Unit 6.	Strength and Failure Theories	B. J. Hartz
Unit 7.	Finite Element Analysis	B. J. Hartz
Unit 8.	Fracture Mechanics of Ceramics	A. S. Kobayashi
Unit 9.	Nondestructive Evaluation	J. L. Bjorkstam
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